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Multi-Media, Multi-Concentration, Organic Analytical Service for Superfund (SOM01.1)

Office of Superfund Remediation and Technology Innovation (OSRTI)
Analytical Services Branch (ASB) (5102G)

Quick Reference Fact Sheet

Under the legislative authority granted to the U.S. Environmental Protection Agency (EPA) under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA), EPA develops standardized analytical methods for the measurement of various pollutants in environmental samples from known or suspected hazardous waste sites. Among the pollutants that are of concern to the EPA at such sites are a series of volatile, semivolatile, pesticide, and Aroclor compounds that are analyzed using gas chromatography coupled with mass spectrometry (GC/MS) and gas chromatography with an electron capture detector (GC/ECD). The Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) offers an analytical service that provides data from the analysis of water and soil/sediment samples for organic compounds for use in the Superfund decision-making process. Through a series of standardized procedures and a strict chain-of-custody, the organic analytical service produces data of known and documented quality. This service is available through the Superfund Contract Laboratory Program (CLP).

DESCRIPTION OF SERVICES

This new organic analytical service provides a technical and contractual framework for laboratories to apply EPA/CLP analytical methods for the isolation, detection, and quantitative measurement of 52 volatile, 67 semivolatile, 21 pesticide, and 9 Aroclor target compounds in water and soil/sediment environmental samples. The CLP provides the methods to be used and the specific technical, reporting, and contractual requirements, including Quality Assurance (QA), Quality Control (QC), and Standard Operating Procedures (SOPs), by which EPA evaluates the data. This service uses GC/MS and GC/ECD methods to analyze the target compounds.

Three data delivery turnarounds are available to CLP customers: 7-day, 14-day, and 21-day turnaround after laboratory receipt of the last sample in the set. In addition, there are 48-hour (for trace volatiles and volatiles) and 72-hour (for semivolatiles, pesticides, and Aroclors) preliminary data submission options available. Changes to the organic method include the separation of pesticide and Aroclor methods, the inclusion of Selected Ion Monitoring (SIM) analysis, and the incorporation of the Staged Electronic Data Deliverable (SEDD) requirement for Electronic Data Deliverables (EDDs) in Extensible Markup Language (XML) format. Options under this service include a closed system purge-and-trap method for low-level volatile soil analysis and methanol preservation for medium-level volatile soil analysis. In addition, data users may request modifications to the SOW that may include, but are not limited to, additional compounds, sample matrices other than soil/sediment or water, lower quantitation limits, and other requirements to enhance method performance.

DATA USES

This analytical service provides data which EPA uses for a variety of purposes, such as determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of a hazardous waste site including, but not limited to: site inspections; Hazard Ranking System (HRS) scoring; remedial investigations/Feasibility Studies (FSs); remedial design; treatability studies; and removal actions. In addition, this service provides data that will be available for use in Superfund enforcement/litigation activities.

TARGET COMPOUNDS

Table 1 lists the compounds for which this service is applicable and the corresponding quantitation limits. Specific quantitation limits are highly matrix-dependent.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.1*

	Quantitation Limits				
	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)
VOLATILES					
1. Dichlorodifluoromethane		0.50	5.0	5.0	250
2. Chloromethane		0.50	5.0	5.0	250
3. Vinyl Chloride		0.50	5.0	5.0	250
4. Bromomethane		0.50	5.0	5.0	250
5. Chloroethane		0.50	5.0	5.0	250
6. Trichlorofluoromethane		0.50	5.0	5.0	250
7. 1,1-Dicholoroethene		0.50	5.0	5.0	250
8. 1,1,2-Trichloro-1,2,2-trifluoroethane		0.50	5.0	5.0	250
9. Acetone		5.0	10	10	500
10. Carbon Disulfide		0.50	5.0	5.0	250
11. Methyl acetate		0.50	5.0	5.0	250
12. Methylene chloride		0.50	5.0	5.0	250
13. trans-1,2-Dichloroethene		0.50	5.0	5.0	250
14. Methyl tert-butyl ether		0.50	5.0	5.0	250
15. 1,1-Dichloroethane		0.50	5.0	5.0	250
16. cis-1,2-Dichloroethene		0.50	5.0	5.0	250
17. 2-Butanone		5.0	10	10	500
18. Bromochloromethane		0.50	5.0	5.0	250
19. Chloroform		0.50	5.0	5.0	250
20. 1,1,1-Trichloroethane		0.50	5.0	5.0	250
21. Cyclohexane		0.50	5.0	5.0	250
22. Carbon tetrachloride		0.50	5.0	5.0	250
23. Benzene		0.50	5.0	5.0	250
24. 1,2-Dichloroethane		0.50	5.0	5.0	250
25. 1,4-Dioxane	2.0	20	100	100	5000
26. Trichloroethene		0.50	5.0	5.0	250
27. Methylcyclohexane		0.50	5.0	5.0	250
28. 1,2-Dichloropropane		0.50	5.0	5.0	250
29. Bromodichloromethane		0.50	5.0	5.0	250
30. cis-1,3-Dichloropropene		0.50	5.0	5.0	250
31. 4-Methyl-2-pentanone		5.0	10	10	500
32. Toluene		0.50	5.0	5.0	250
33. trans-1,3-Dichloropropene		0.50	5.0	5.0	250
34. 1,1,2-Trichloroethane		0.50	5.0	5.0	250
35. Tetrachloroethene		0.50	5.0	5.0	250
36. 2-Hexanone		5.0	10	10	500
37. Dibromochloromethane		0.50	5.0	5.0	250
38. 1,2-Dibromoethane	0.050	0.50	5.0	5.0	250
39. Chlorobenzene		0.50	5.0	5.0	250

	Quantitation Limits				
	Trace Water by SIM (µg/L)	Trace Water (µg/L)	Low Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)
VOLATILES (CON'T)					
40. Ethylbenzene		0.50	5.0	5.0	250
41. o-Xylene		0.50	5.0	5.0	250
42. m, p-Xylene		0.50	5.0	5.0	250
43. Styrene		0.50	5.0	5.0	250
44. Bromoform		0.50	5.0	5.0	250
45. Isopropylbenzene		0.50	5.0	5.0	250
46. 1,1,2,2-Tetrachloroethane		0.50	5.0	5.0	250
47. 1,3-Dichlorobenzene		0.50	5.0	5.0	250
48. 1,4-Dichlorobenzene		0.50	5.0	5.0	250
49. 1,2-Dichlorobenzene		0.50	5.0	5.0	250
50. 1,2-Dibromo-3-chloropropane	0.050	0.50	5.0	5.0	250
51. 1,2,4-Trichlorobenzene		0.50	5.0	5.0	250
52. 1,2,3-Trichlorobenzene		0.50	5.0	5.0	250
SEMIVOLATILES	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
53. Benzaldehyde		5.0		170	5000
54. Phenol		5.0		170	5000
55. bis-(2-chloroethyl) ether		5.0		170	5000
56. 2-Chlorophenol		5.0		170	5000
57. 2-Methylphenol		5.0		170	5000
58. 2,2'-Oxybis (1-chloropropane)		5.0		170	5000
59. Acetophenone		5.0		170	5000
60. 4-Methylphenol		5.0		170	5000
61. N-Nitroso-di-n propylamine		5.0		170	5000
62. Hexachloroethane		5.0		170	5000
63. Nitrobenzene		5.0		170	5000
64. Isophorone		5.0		170	5000
65. 2-Nitrophenol		5.0		170	5000
66. 2,4-Dimethylphenol		5.0		170	5000
67. Bis (2-chloroethoxy) methane		5.0		170	5000
68. 2,4-Dichlorophenol		5.0		170	5000
69. Napthalene	0.10	5.0	3.3	170	5000
70. 4-Chloroaniline		5.0		170	5000
71. Hexachlorobutadiene		5.0		170	5000
72. Caprolactam		5.0		170	5000
73. 4-Chloro-3-methylphenol		5.0		170	5000
74. 2-Methylnapthalene	0.10	5.0	3.3	170	5000
75. Hexachlorocyclopentadiene		5.0		170	5000
76. 2,4,6-Trichlorophenol		5.0		170	5000
77. 2,4,5-Trichlorophenol		5.0		170	5000

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 50 times the quantitation limits for low soils.

Table 1. Target Compound List (TCL) and Contract Required Quantitation Limits (CRQLs) for SOM01.1* (Con't)

	Quantitation Limits				
	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
SEMIVOLATILES (CON'T)					
78. 1,1'-Biphenyl		5.0		170	5000
79. 2-Chloronapthalene		5.0		170	5000
80. 2-Nitroaniline		10		330	10000
81. Dimethylphthalate		5.0		170	5000
82. 2,6-Dinitrotoluene		5.0		170	5000
83. Acenaphthylene	0.10	5.0	3.3	170	5000
84. 3-Nitroaniline		10		330	10000
85. Acenaphthene	0.10	5.0	3.3	170	5000
86. 2,4-Dinitrophenol		10		330	10000
87. 4-Nitrophenol		10		330	10000
88. Dibenzofuran		5.0		170	5000
89. 2,4-Dinitrotoluene		5.0		170	5000
90. Diethylphthalate		5.0		170	5000
91. Fluorene	0.10	5.0	3.3	170	5000
92. 4-Chlorophenyl phenyl ether		5.0		170	5000
93. 4-Nitroaniline		10		330	10000
94. 4,6-Dinitro-2-methylphenol		10		330	10000
95. N-Nitrosodiphenylamine		5.0		170	5000
96. 1,2,4,5-Tetrachlorobenzene		5.0		170	5000
97. 4-Bromophenyl phenyl ether		5.0		170	5000
98. Hexachlorobenzene		5.0		170	5000
99. Atrazine		5.0		170	5000
100. Pentachlorophenol	0.20	10	6.7	330	10000
101. Phenanthrene	0.10	5.0	3.3	170	5000
102. Anthracene	0.10	5.0	3.3	170	5000
103. Carbazole		5.0		170	5000
104. Di-n-butylphthalate		5.0		170	5000
105. Fluoranthene	0.10	5.0	3.3	170	5000
106. Pyrene	0.10	5.0	3.3	170	5000
107. Butylbenzylphthalate		5.0		170	5000
108. 3,3'-Dichlorobenzidine		5.0		170	5000
109. Benzo(a)anthracene	0.10	5.0	3.3	170	5000
110. Chrysene	0.10	5.0	3.3	170	5000
111. Bis(2-ethylhexyl)phthalate		5.0		170	5000
112. Di-n-octylphthalate		5.0		170	5000
113. Benzo(b)fluoroanthene	0.10	5.0	3.3	170	5000
114. Benzo(k)fluoroanthene	0.10	5.0	3.3	170	5000

	Quantitation Limits				
	Low Water by SIM (µg/L)	Low Water (µg/L)	Low Soil by SIM (µg/kg)	Low Soil (µg/kg)	Med. Soil (µg/kg)
SEMIVOLATILES (CON'T)					
115. Benzo(a)pyrene	0.10	5.0	3.3	170	5000
116. Indeno(1,2,3-cd)pyrene	0.10	5.0	3.3	170	5000
117. Dibenzo(a,h)anthracene	0.10	5.0	3.3	170	5000
118. Benzo(g,h,i)perylene	0.10	5.0	3.3	170	5000
119. 2,3,4,6-Tetrachlorophenol		5.0		170	5000
PESTICIDES	**Water (µg/L)**			**Soil (µg/kg)**	
120. alpha-BHC	0.050			1.7	
121. beta-BHC	0.050			1.7	
122. delta-BHC	0.050			1.7	
123. gamma-BHC (Lindane)	0.050			1.7	
124. Heptachlor	0.050			1.7	
125. Aldrin	0.050			1.7	
126. Heptachlor epoxide	0.050			1.7	
127. Endosulfan I	0.050			1.7	
128. Dieldrin	0.10			3.3	
129. 4,4'-DDE	0.10			3.3	
130. Endrin	0.10			3.3	
131. Endosulfan II	0.10			3.3	
132. 4-4'-DDD	0.10			3.3	
133. Endosulfan sulfate	0.10			3.3	
134. 4-4'-DDT	0.10			3.3	
135. Methoxychlor	0.50			17	
136. Endrin ketone	0.10			3.3	
137. Endrin aldehyde	0.10			3.3	
138. alpha-Chlordane	0.050			1.7	
139. gamma-Chlordane	0.050			1.7	
140. Toxaphene	5.0			170	
AROCLORS	**Water (µg/L)**			**Soil (µg/kg)**	
141. Aroclor-1016	1.0			33	
142. Aroclor-1221	1.0			33	
143. Aroclor-1232	1.0			33	
144. Aroclor-1242	1.0			33	
145. Aroclor-1248	1.0			33	
146. Aroclor-1254	1.0			33	
147. Aroclor-1260	1.0			33	
148. Aroclor-1262	1.0			33	
149. Aroclor-1268	1.0			33	

* For volatiles, quantitation limits for medium soils are approximately 50 times the quantitation limits for low soils. For semivolatile medium soils, quantitation limits are approximately 30 times the quantitation limits for low soils.

The TCL for this service was originally derived from the EPA Priority Pollutant List of 129 compounds. In the years since inception of the CLP, compounds have been added to and removed from the TCL, based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. The SOM analytical service combines the previous OLM and OLC services into one method. For example, drinking water and ground water type samples may be analyzed using the Trace Volatiles method in SOM.

METHODS AND INSTRUMENTATION

For trace volatile water samples, 25 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. For low/medium volatile water samples, 5 mL of water sample is added to a purge-and-trap device and purged with an inert gas at room temperature. Higher purge temperatures may be used for both trace and low/medium volatile analyses if all technical acceptance criteria is met for all standards, samples, and blanks. For low-level volatile soil samples, organic compounds are generally determined by analyzing approximately 5 g of sample in a closed-system purge-and-trap device at 40°C. For a medium-level soil sample, a soil sample of 5 g is collected, preserved, and/or extracted with methanol and an aliquot of methanol extract is added to 5 mL reagent water and purged at room temperature. For water and soil samples, the volatiles purged from the sample are trapped on a solid sorbent. The purged volatiles are subsequently desorbed by rapidly heating and backflushing with helium, and then introduced into a GC/MS system.

For semivolatile, pesticide, and Aroclor water samples, a 1 L aliquot of sample is extracted with methylene chloride using a continuous liquid-liquid extractor or separatory funnel (for pesticides and Aroclors only). For low-level semivolatile, pesticide, and Aroclor soil samples, a 30 g soil/sediment sample is extracted with methylene chloride/acetone using sonication, automated Soxhlet/Dean-Stark (SDS) extraction, or pressurized fluid extraction techniques. For medium-level semivolatile soil samples, a 1g aliquot is extracted with methylene chloride using the techniques mentioned above for low-level soil samples. For both water and soil samples, the extract is concentrated, subjected to fraction-specific cleanup procedures, and analyzed by GC/MS for semivolatiles or GC/ECD for pesticides and Aroclors. **Table 2** summarizes the methods and instruments used in this analytical service.

DATA DELIVERABLES

Data deliverables for this service include hardcopy data reporting forms and supporting raw data. In addition to the hardcopy deliverable, contract laboratories must also submit the same data electronically. The laboratory must submit data to EPA within 7, 14, or 21-days after laboratory receipt of the last sample in set [or preliminary data within 48 hours (for trace volatiles and volatiles) or 72 hours (for semivolatiles, pesticides, and Aroclors) after laboratory receipt of each sample. EPA then processes the data through an automated Data Assessment Tool (DAT). DAT provides EPA Regions with PC-compatible reports, spreadsheets, and electronic files within 24-48 hours from the receipt of the data for use in data validation. This automated tool also facilitates the transfer of analytical data into Regional databases. In addition to the Regional electronic reports, the CLP laboratories are provided with a data assessment report that documents the instances of noncompliance. The laboratory has 6 business days to reconcile defective data and resubmit the data to EPA. EPA then reviews the data for noncompliance and sends a final data assessment report to the CLP laboratory and the Region.

QUALITY ASSURANCE (QA)

The QA process consists of management review and oversight at the planning, implementation, and completion stages of the environmental data collection activity. This process ensures that the data provided are of known and documented quality.

During the implementation of the data collection effort, QA activities ensure that the Quality Control (QC) system is functioning effectively and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data to determine its suitability to support enforcement or remedial decisions.

Each contract laboratory prepares a Quality Assurance Plan (QAP) with the objective of providing sound analytical chemical measurements. The QAP must specify the policies, organization, objectives, and functional guidelines, as well as the QA and QC activities designed to achieve the data quality requirements in the contract.

QUALITY CONTROL (QC)

The QC process includes those activities required during analytical data collection to produce data of known and documented quality. The analytical data acquired from QC procedures are used to estimate and evaluate the analytical results and to determine the necessity for, or the effect of, corrective action procedures. The QC procedures required for this analytical service are provided in **Table 3.**

Table 2. Methods and Instruments

Fraction	Water	Soil
Trace Volatiles	Purge-and-trap followed by GC/MS analysis	N/A
Volatiles	Purge-and-trap followed by GC/MS analysis	Purge-and-trap or closed-system purge-and-trap followed by GC/MS analysis
Semivolatiles	Continuous liquid-liquid extraction (CLLE) followed by GC/MS analysis	Sonication, automated SDS extraction, or pressurized fluid extraction followed by GC/MS analysis
Pesticides	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis
Aroclors	CLLE or separatory funnel extraction followed by dual column GC/ECD analysis	Sonication, automated SDS extraction or pressurized fluid extraction followed by dual column GC/ECD analysis

Table 3. Quality Control (QC)

QC Operation	Frequency
Deuterated Monitoring Compounds (DMCs) (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Surrogates (pesticides and Aroclors)	Added to each sample, standard, and blank
Method Blanks (trace volatiles and volatiles)	Analyzed at least every 12 hours for each matrix and level
Method Blanks (semivolatiles, pesticides, and Aroclors)	Prepared with each group of 20 samples or less of same matrix and level, or each time samples are extracted by the same procedure
Instrument Blank (trace volatiles and volatiles)	Analyzed after a sample which contains compounds at concentrations greater than the calibration range
Instrument Blank (pesticides and Aroclors)	Every 12 hours on each GC column used for analysis
Storage Blanks (trace volatiles and volatiles)	Prepared and stored with each set of samples
GC/MS Mass Calibration and Ion Abundance Patterns (trace volatiles, volatiles, and semivolatiles)	Every 12 hours for each instrument used for analysis
GC Resolution Check (pesticides)	Prior to initial calibration, on each instrument used for analysis
Initial Calibration	Upon initial set up of each instrument, and each time continuing calibration fails to meet the acceptance criteria
Continuing Calibration	Every 12 hours for each instrument used for analysis
Internal Standards (trace volatiles, volatiles, and semivolatiles)	Added to each sample, standard, and blank
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	Once every 20 or fewer samples of same fraction, matrix, and level in a Sample Delivery Group (SDG)
Laboratory Control Samples (LCSs) (pesticides and Aroclors)	Once every 20 or fewer samples of same fraction, matrix, and level in an SDG
Method Detection Limit (MDL)	Determined annually, per matrix and level

PERFORMANCE MONITORING ACTIVITIES

Laboratory performance monitoring activities are provided primarily by ASB and the Regions to ensure that contract laboratories are producing data of the appropriate quality. EPA performs on-site laboratory audits, data package audits, GC/MS and/or GC/ECD tape audits, and evaluates laboratory performance through the use of blind Performance Evaluation (PE) samples.

CONTACTING EPA

For more information, or for suggestions to improve this analytical service, please contact:

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